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NON-ADIABATIC CAPTURE OF MOBILE ELECTRONS IN AQUEOUS SOLUTION.(U)

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⁶ Non-adiabatic Capture of Mobile Electrons in Aqueous Solution •

by

¹⁰ Kathrin von Burg and Paul Delahay

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NON-ADIABATIC CAPTURE OF MOBILE ELECTRONS IN AQUEOUS SOLUTION

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Experimental evidence and theoretical arguments are presented for 11 effective and eight ineffective cations for non-adiabatic capture of mobile electrons in the photoionization (< 10.5 eV) of anions (Cl^- , ClO_4^- , SO_4^{2-}) in aqueous solution. This effect lowers yields and increases threshold energies for photoelectron emission by anions into the gas phase.

1. Introduction

The yield for photoelectron emission by aqueous solutions of inorganic anions (Cl^- , SO_4^{2-}) was recently observed [1] to be lower in the presence of certain cations (e.g., H^+ , Fe^{2+}) than for the corresponding salts of the alkali metals. The effect is displayed in fig. 1. Curve A (2 M KCl) shows the emission by Cl^- (threshold energy $E_t = 8.77$ eV [2]), and B (1 M FeCl_2) displays the emission by Fe^{2+} ($E_t = 7.35$ eV [1]) and, at higher photon energies ($E \approx 9$ eV), by Cl^- . Since the Cl^- concentration is the same for A and B, one would expect higher yields for B than A over the full range of photon energies. The opposite is observed for $E \approx 9.0$ eV, that is, in the range in which emission by Cl^- becomes significant. Moreover, it was found [1] that the threshold energy E_t of Cl^- for the FeCl_2 solution is higher (~ 9.1 eV) than E_t of Cl^- for the KCl solution (8.77 eV [2]). The effect of cations on emission by anions was only referred to incidentally in [1] but was investigated in detail later. The results of this new work are discussed here.

2. Experimental results

Emission spectra displaying the yield Y against photon energy E (< 10.5 eV) were obtained by the methods reported in [3] and applied in

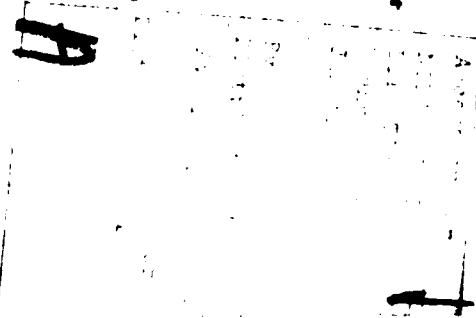
[1,2]. Two cases were distinguished in the analysis of data:

(i) The cation (e.g., H^+ , K^+) does not emit electrons for $E \leq 10.5$ eV. Plots of Y^n vs. E [4] were prepared with $n = 0.5$ [2] for the anions (Cl^- , ClO_4^- , SO_4^{2-}) studied here. Linearity of these plots was apparently not affected by the effect of cations on emission by anions. The threshold energy E_t of the anion was determined by extrapolation to $Y = 0$.

(ii) Both the cations C and anions A emit ($E_t(C) < E_t(A)$). Plots of Y^n vs. E were prepared in the range ($E < E_t(A)$) in which only cations emit ($Y \equiv Y(C)$), and $E_t(C)$ was obtained by extrapolation to $Y = 0$. Selection of n for cations is discussed in [1]. The resulting linear plots were extended to $E > E_t(A)$, and $Y(A) = Y - Y(C)$ was computed. The threshold energy $E_t(A)$ was obtained by extrapolation to $Y(A) = 0$ from the plot of $[Y(A)]^n$ vs. E ($n = 0.5$).

The following cases will be considered: (i) Only the anion emits electrons, and the cation either affects (H^+ , Ba^{2+}) emission by the anion or does not (e.g., K^+). (ii) Both the anion and cation (e.g., Fe^{2+}) display photoelectron emission, and the cation affects emission by the anion.

Results at constant anion concentration will be discussed first for $HCl + KCl$ and $H_2SO_4 + Li_2SO_4$ mixtures (no emission by cations). Since Y at a given E is proportional to the anion concentration [1-4], the squared slope of the $Y^{0.5}$ vs. E plot, $(dY^{0.5}/dE)^2$, should be proportional to the anion concentration. The latter is constant in the present experiments, and therefore the squared slope should be constant in the absence of a cation effect. Actually, the squared slope decreased linearly with increasing H^+ concentration, x , and the threshold energies of Cl^- and SO_4^{2-} shift to higher values (fig. 2).



Similar experiments were done with $\text{MCl}_2 + \text{KCl}$ mixtures at constant Cl^- concentration (2 M) for Mg^{2+} , Ca^{2+} , Sr^{2+} , Ba^{2+} (no emission by cations). Except for Ba^{2+} , no evidence was found for a cation effect on emission by Cl^- . The squared slope and threshold energy ($E_t = 8.77 \pm 0.05$ eV) were independent of M^{2+} ion concentration. Results for BaCl_2 were similar to those of fig. 2: ratio of squared slopes equal to 0.50 for 1 M BaCl_2 /2 M KCl , $\Delta E_t(1 \text{ M BaCl}_2) \approx 0.25$ eV. Thus, Ba^{2+} ions affect the emission by Cl^- . A similar conclusion was reached for emission by ClO_4^- (fig. 3) over a wider concentration range than allowed by BaCl_2 solubility.

The shift of threshold energy ($E_t = 8.77$ and 8.65 eV for Cl^- and SO_4^{2-} , respectively [2]) resulting from the cation effect was determined for several cations (1 M solutions) studied in [1]: $\Delta E_t(\text{Cl}^-) \approx 0.40$ eV for Cr^{2+} , Mn^{2+} , Co^{2+} , Ni^{2+} , V^{3+} , Cr^{3+} ; $\Delta E_t(\text{SO}_4^{2-}) \approx 0.34$ eV for V^{2+} , Cr^{2+} , Ni^{2+} , Cu^{2+} . The standard deviation for these values of ΔE_t varied from 0.05 to 0.15 eV. A more detailed study was made for $\text{FeCl}_2 + \text{KCl}$ mixtures at constant Cl^- concentration (2 M). Results were similar to those of fig. 2: ratio of squared slopes equal to 0.45 for 1M FeCl_2 /2 M KCl , $\Delta E_t(\text{Cl}^-) = 0.28$ eV for 1 M FeCl_2 .

The dependence of $Y(\text{Cl}^-)$ on concentration was also investigated with pure salts and pure HCl . The squared slope $(d[Y(\text{Cl}^-)]^{0.5}/dE)^2$ should be proportional to the salt (or HCl) concentration in the absence of cation effect [1-4]. Proportionality was verified for the alkali cations and the NH_4^+ ions (no cation effect) but was not observed for HCl and FeCl_2 . Curves with a maximum were observed in these two cases (fig. 4). Emission by Fe^{2+} was not affected in any way similar to emission by anions. The quantity $(d[Y(\text{Fe}^{2+})]^{0.4}/dE)^{2.5}$ ($n = 0.4$ for Fe^{2+} [1]) increased linearly with FeCl_2 concentration (fig. 4), and E_t (7.35 eV) was independent of this concentration. Similar observations to those for Fe^{2+} were made for the

cations studied in [1]. There was also no significant change of the threshold energies of Ag^+ , Tl^+ , Sn^{2+} , Pb^{2+} , Fe^{3+} when solutions of these cations were made highly acidic (up to 6 M).

3. Non-adiabatic capture of mobile electrons by cations

Two different approaches can be followed to interpret the results of sec. 2. (i) One supposes that the photoionization rate of anions is decreased by cations, e.g., because of complexation of anions or attenuation of the photon flux in solution. Such an interpretation could not be reconciled with experiment and was ruled out. (ii) One assumes that photoionization of anions is not affected by cations, but that some of the electrons generated by this process are captured by cations. This second interpretation is adopted here.

Electrons upon emission from solution into the gas phase exhibit a distribution over the full range of kinetic energies which is consistent with energy conservation for the prevailing threshold and photon energies [5,6]. A major decrease in yield would not be expected under these conditions if capture was restricted to thermalized and/or hydrated electrons. Yet major lowering in yield is observed, e.g., the ratio of squared slopes for HCl/LiCl is equal to 0.14 for 6 M solutions. We therefore conclude that lowering of the emission yield cannot be attributed to the loss of electrons which were thermalized in solution prior to capture by cations.

Capture by different cations M^{2+} will be examined by comparing the standard reduction potential E^0 for M^{2+}/M^+ or M^+/M with the energy level of mobile electrons. The E^0 's (in V) are [7]: ~ -3.5 (Na^+ , K^+ , Cs^+), -4.0 to -5.5 (Mg^{2+} , Ca^{2+} , Sr^{2+} , Ba^{2+}), -2.7 to -3.1 (Mn^{2+} , Fe^{2+} , Co^{2+} , Ni^{2+}). The bottom of the conduction band of mobile electrons in water is ~ 0.25 V more negative [8,9] in the hydrogen electrode scale than the standard potential

of the hydrated electron (-2.77 eV). Hence, one has $E^m \leq -3.0$ V for mobile electrons. On the basis of the E^0 and E^m values, one concludes that capture should be possible for the transition metal cations M^{2+} but impossible for Ba^{2+} . Yet Ba^{2+} ($E^0 \approx -4.6$ to -5.1 V) ions exhibit a cation effect whereas the alkali metal cations having lower $|E^0|$ values (~ -3.5 V) than Ba^{2+} do not. Thus, the foregoing analysis leads to contradictions.

The difficulties with the previous interpretation are removed if capture is considered in a time interval which is so short that only electronic polarization of the medium about the cation changes upon capture of a mobile electron. The species generated in such a non-adiabatic process (in the spectroscopic sense) should ultimately be reoxidized (e.g., by H^+) after relaxation of the orientation polarization. The difference $\Delta G_S^2 - \Delta G_S^1$ between the solvation energies of M^{2+} and M^+ appearing in the expression of E^0 for M^{2+}/M^+ [1,7] is smaller in the non-adiabatic case than for the adiabatic process. The difference $\Delta G_S^2 - \Delta G_S^1$ can be calculated, at least approximately, by means of the Born equation and by using the optical dielectric constant of water. One obtains (ionic radii from [10], other data from [11]) the potential (in V) $E^* = -3.9$ (Mg^{2+}), -2.2 (Ca^{2+}), -1.8 (Sr^{2+}), -1.4 (Ba^{2+}), -1.0 (V^{2+}), -0.8 (Fe^{2+}), -0.7 (Mn^{2+}), and less negative values for Cr^{2+} , Co^{2+} , Ni^{2+} , Cr^{3+} , H^+ . One also has $E^* \leq -2.5$ V for the alkali metal cations. These values of E^* are in general much less negative than the corresponding E^0 's for an adiabatic process.

The sequence of E^* values agrees with the results in sec. 2: cation effect for $E^* \geq -1.4$ V (Ba^{2+}) and no effect for $E^* \leq -1.8$ V (Sr^{2+}). These two limits for E^* cannot be compared directly with the value $E^m \leq -3.0$ V for mobile electrons. Thus, the orientation polarization of the medium about the anion emitting an electron does not change in the time interval

before capture of this electron by a cation. The electron therefore is in the potential well resulting from the orientation polarization about the anion. The potential energy is [12] $-(e^2/r)(\epsilon_0^{-1} - \epsilon_s^{-1})$, where e is the electronic charge, r the distance from the center of the well, ϵ_0 and ϵ_s the optical and static dielectric constants of water, respectively. If one assumes, for instance, a distance of 0.5 nm between anion and cation in solution, the potential energy is ~ -1.6 eV. Hence, the potential to be compared with the E^* values is not $E^m \sim -3.0$ V but rather $E \sim -1.4$ V. The limit of -1.4 V corresponds to $E^* = -1.4$ V for Ba^{2+} . A quantum [12] rather than classical calculation should be performed, and the uncertainty on energy on the scale of time being considered may not be negligible. Anyhow, our analysis does show that the process of non-adiabatic capture being proposed seems justified.

The foregoing analysis does not account for the absence of cation effect on the emission by cations (e.g., Fe^{2+} , sec. 2). Low-energy mobile electrons have a thermalization length (2 to 4 nm [9,13]) which exceeds the average distance between ions of the same sign (≈ 1.2 nm for 1 M). Capture of mobile electrons by cations prior to thermalization therefore seems possible in the photoionization of either cations or anions. The same conclusion is reached from consideration of the de Broglie wavelength of electrons [14] rather than their thermalization length. Actually, the threshold energies of cations are independent of their concentration (e.g., Fe^{2+}). Thus, the capture of mobile electrons must somehow be restricted to shorter distances than the thermalization length. This would be the case if capture occurs via tunneling [14,15]. Moreover, the average ion configurations are different for emission by anions and cations: emitting anions are surrounded by electron-capturing cations whereas emitting cations are shielded by anions from the nearest cations.

4. Kinetics of electron capture

The results of fig. 2 to 4 can be interpreted in a simple way by assuming that the generation of electrons and their capture proceed at rates which are proportional, respectively, to the anion (C_a) and cation (C_c) concentrations. If C_a is constant, the production rate is constant but the capture rate and consequently the emission yield decrease with increasing concentration C_c (fig. 2 and 3). If $C_a = C_c$ (HCl) or $C_a = 2C_c$ ($FeCl_2$), a maximum in the curve of the squared slope vs. C_c is observed (fig. 4) because capture overtakes production. The ratio of the squared slope to concentration for the data on HCl and $FeCl_2$ of fig. 4 decreases linearly with concentration up to ~ 1.5 M Cl^- concentration and more slowly at higher concentrations. A similar deviation from simple homogeneous kinetics is observed for the capture of mobile electrons in radiolysis studies of liquids [14]. The deviation can be interpreted as indicative of tunneling [14].

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Captions to Figures

Fig. 1. Photoelectron emission spectra of 2 M KCl (A) and 1 M FeCl_2 (B).

Fig. 2. Squared slope (top) vs. concentration x for x M HCl + $(2 - x)$ M KCl (A) and $x/2$ M H_2SO_4 + $(1 - x/2)$ M Li_2SO_4 (B). Shift ΔE_t of threshold energy (bottom) vs. x for emission by Cl^- (A) and SO_4^{2-} (B). $E_t = 8.77$ and 8.65 eV, respectively, for 2 M KCl and 1 M Li_2SO_4 .

Fig. 3. Squared slope (top) vs. concentration x for x M $\text{Ba}(\text{ClO}_4)_2$ + $(6 - 2x)$ M NaClO_4 and shift ΔE_t for ClO_4^- vs. x (bottom). $E_t = 8.45$ for ClO_4^- .

Fig. 4. Squared slope for emission by Cl^- ions vs. concentration of HCl (A) and FeCl_2 (B). Slope to the power 2.5 for emission by Fe^{2+} ions vs. concentration of FeCl_2 (C).

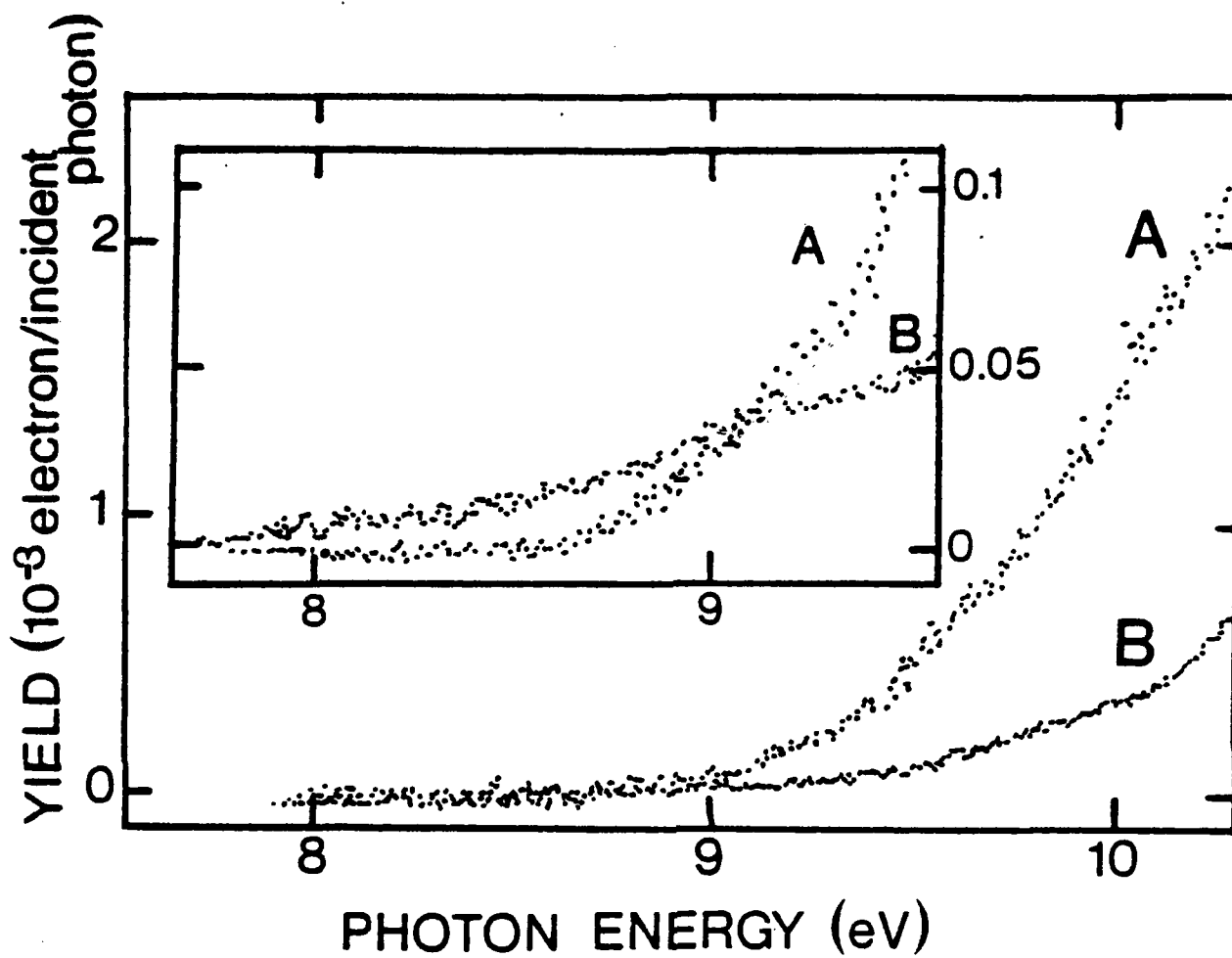


FIG. 1

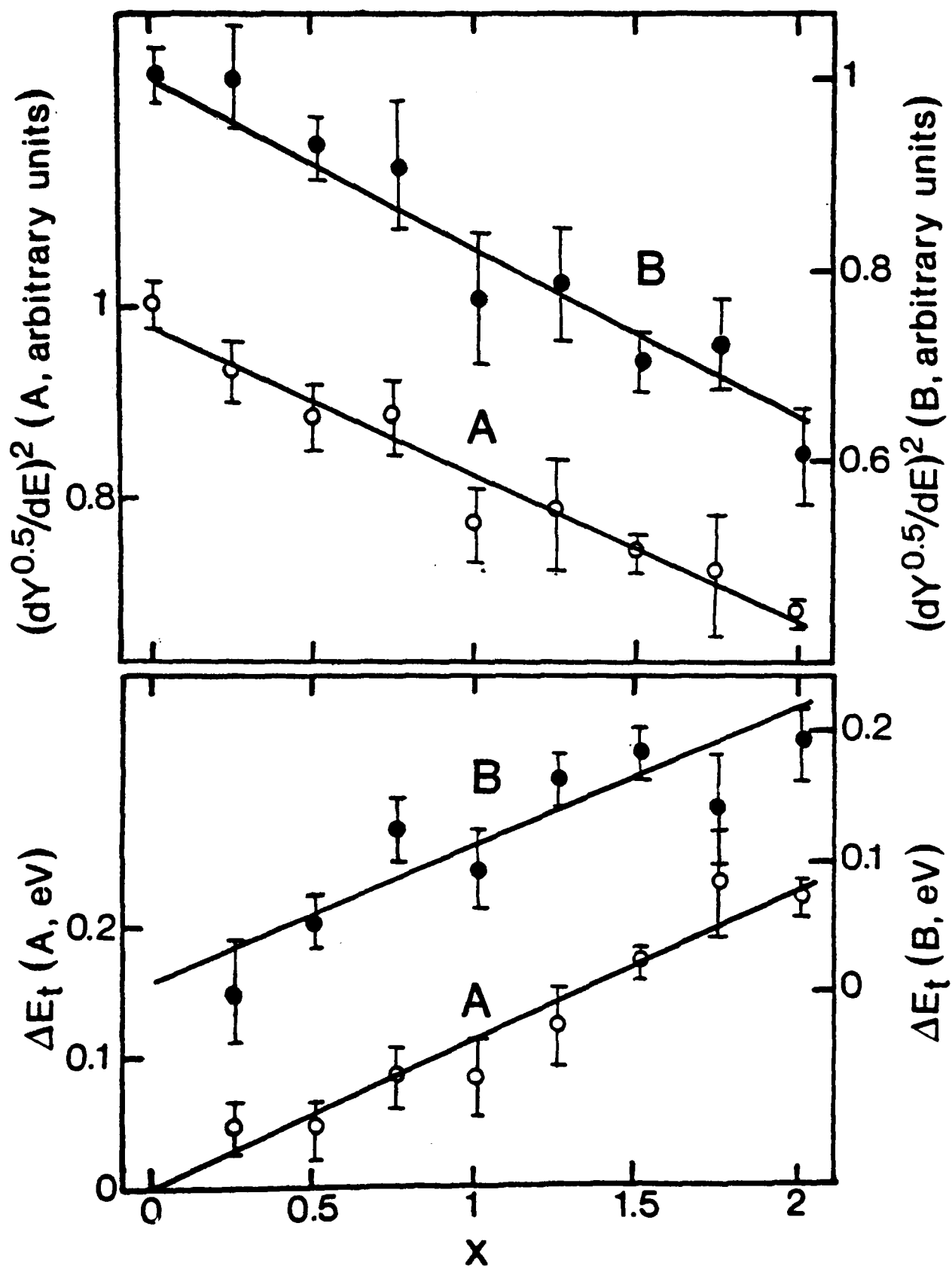


FIG. 2

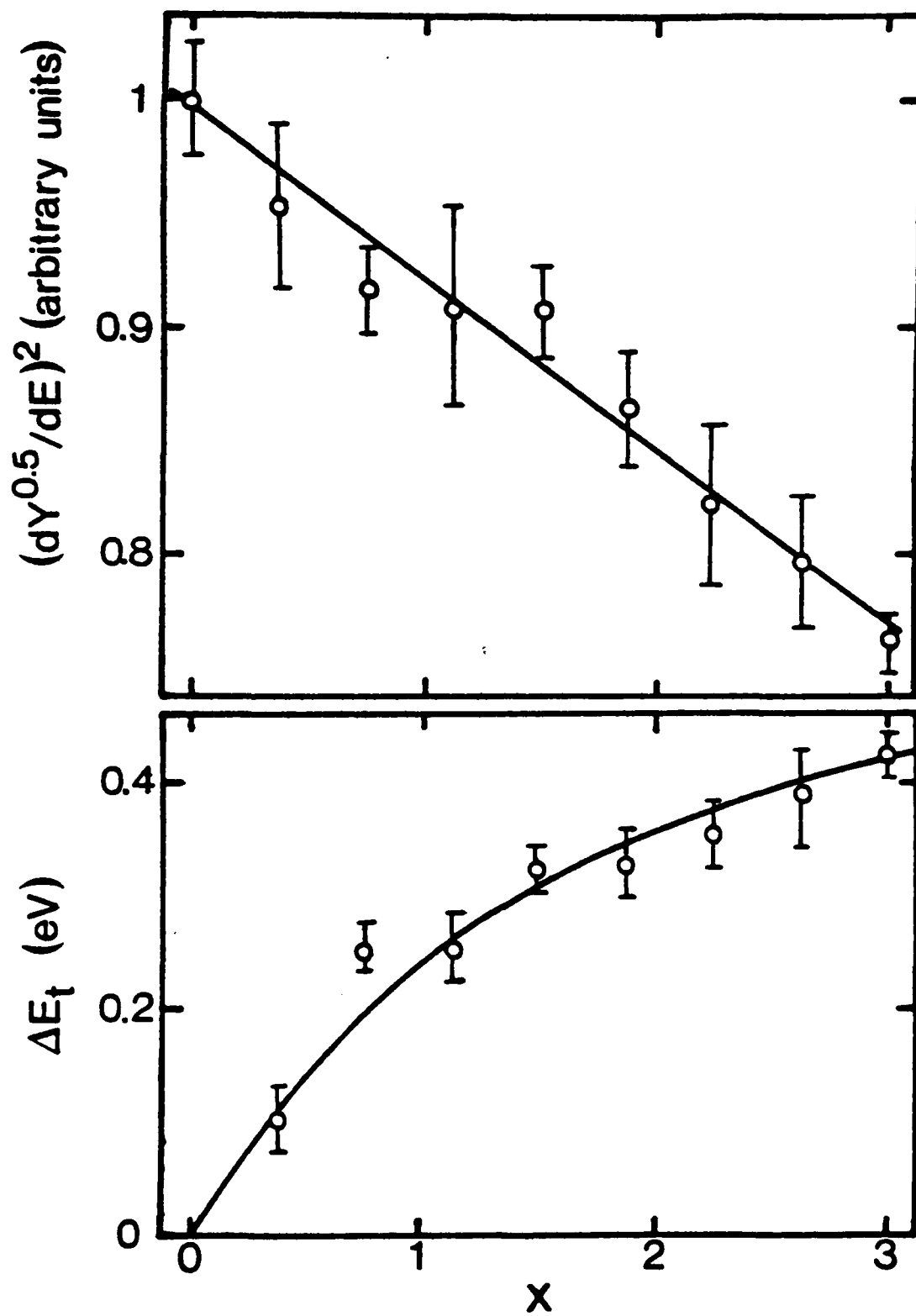


Fig. 3

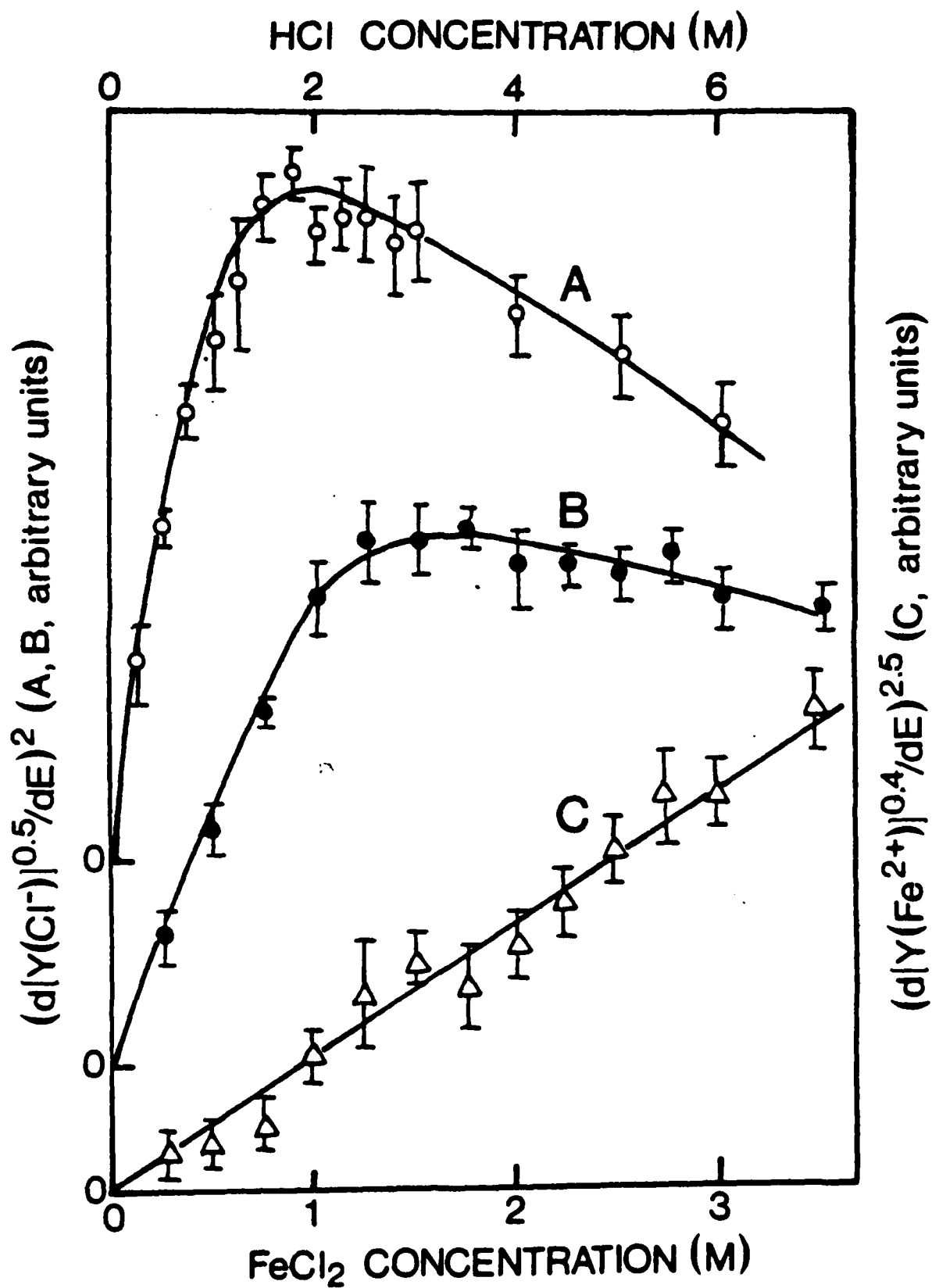


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